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DELIVERY OF MENTHOL FROM CIGARETTES CONTAINING EITHER A MENTHOLATED FILTER OR MENTHOLATED TOBACCO

John G. Curran

Research Laboratories/Tennessee Eastman Company

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INTRODUCTION

The increased popularity of mentholated cigarettes has necessitated a better understanding of what happens to menthol after it has been added to a cigarette. Newell et al.[1] reported that approximately 70% of the menthol injected into a filter cigarette was recovered, and only a small amount was destroyed in the burning zone of a cigarette. Dawson et al.[2] reported that 98.9% of total mainstream activity produced from tobacco treated with C^{14}-labeled menthol was attributed to C^{14}-labeled menthol.

Evaluations conducted in the Research Laboratories at Tennessee Eastman Company, a division of Eastman Kodak Company, were concerned with filter cigarettes constructed with either mentholated tobacco or mentholated filters. The objectives of these studies were to determine the following: whether menthol migrates from the filter to the tobacco and from the tobacco to the filter; whether the age of the cigarette or the initial placement of menthol (tobacco or filter) influences the delivery of menthol.

EXPERIMENTAL

Mentholated cigarettes (12 hr old) were stored for different lengths of time and then smoked or separated into tobacco and filter sections. The total particulate matter (TPM) from individual puffs and from whole cigarettes was collected. The menthol in the TPM and in different cigarette sections (tobacco and filter) was determined by gas chromatography.

Materials and Methods

Cigarettes — A mentholated cigarette and its nonmentholated equivalent were obtained from their manufacturer 12 hr after they were produced. Cigarette T, a commercially available cigarette, contained tobacco treated with menthol (65-mm tobacco column) and a 20-mm cellulose acetate filter. Cigarette F, which is not commercially available, was constructed from a 65-mm tobacco column of a nonmentholated cigarette and a 20-mm cellulose acetate filter. The tow of the filter was bonded with a blend of 55% triacetin and 45% polyethylene glycol treated with menthol, 26% w/w.

Storage and Testing of Cigarettes — Several packs of cigarettes T and F were placed in storage. Storage time varied less than ± 1 hr for the stipulated number of days. In migration experiments, cigarettes T and F were taken from storage after 1, 2, 3, 6, 7, 8 9, 14 and 28 days and separated into two segments — filter and tobacco — before being analyzed. In smoking experiments, cigarettes T and F, 3 and 28 days old, were smoked by use of a Phipps and Bird automated smoking machine[a] in accordance with Federal Trade Commission specifications. TPM from three cigarettes was collected on a Cambridge filter pad[b], and each pad was analyzed. In individual puff experiments, the TPM for each puff was collected on a separate Cambridge filter pad. A cigarette was placed in a port and lighted. At the completion of the puff, the cigarette was removed; a clearing puff of air was drawn through the port, and the cigarette was placed in another port in preparation for the second puff. This sequence was repeated until nine puffs had been taken from each cigarette, and a total of ten cigarettes had been smoked.

Analysis — Methanol was used to extract menthol from the three cellulose acetate filters, the Cambridge filter pad, and the three tobacco columns. A known weight of internal standard (anethole) was added to each extract. The extracts were shaken for 1 hr before being analyzed by gas chromatography. The gas chromatograph was equipped with a single 4-ft × ⅛-in. stainless steel column with Chromosorb W packing[c] (60-80 mesh) coated with 14% w/w diethylene glycol succinate. Operating conditions were as follows: injector and detector temperature 180°C; column temperature programmed at 4°C per minute from 130 to 170°C; helium flow 63 ml/minute; hydrogen flow 22 ml/minute; air flow 350 ml/minute.

Accuracy and Precision of Analytical Technique — Tobacco columns from nonmentholated cigarettes and filters and Cambridge filter pads containing TPM from nonmentholated cigarettes were placed in separate vials, and 0.83 mg menthol/ml methanol was added to each of these materials. The menthol/methanol solution was added so that all the liquid was sorbed by the material. The vials were sealed for 1 hr before the menthol was extracted and analyzed. This procedure was repeated five times for each material, and a sample of the mentholated tobacco was analyzed ten separate times to determine the precision of the method.

RESULTS AND DISCUSSION

Accuracy and Precision of Analytical Technique

Tables I and II show that the accuracy and precision of the analytical technique were adequate for quantitative determinations of menthol on tobacco, cellulose acetate filters, and Cambridge filter pads.

TABLE I
ACCURACY OF MENTHOL ANALYSIS

Run	Menthol Recovered, %		
	Filter[d]	Tobacco[d]	Cambridge Filter Pad[d]
1	98.8	101.2	97.6
2	98.8	102.4	96.4
3	100.0	98.8	97.6
4	97.6	100.0	97.6
5	98.8	103.6	97.6
$\bar{X}$	98.8	101.2	97.4
S	0.8	1.9	0.5
CV	0.8	1.9	0.6

[d]These materials were treated with 0.83 mg menthol.

TABLE II
PRECISION OF MENTHOL ANALYSIS

Run	Menthol, mg
1	0.54
2	0.55
3	0.55
4	0.54
5	0.54
6	0.53
7	0.55
8	0.52
9	0.53
10	0.54
$\bar{X}$	0.54
S	0.01
CV	1.9

[a]Phipps and Bird, Inc., Richmond, Virginia
[b]Cambridge Filter Company, Syracuse, New York
[c]Johns-Manville Corporation, New York, New York

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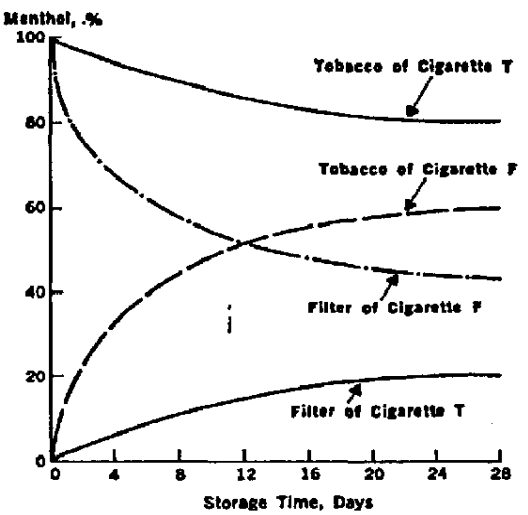
Migration of Menthol in a Cigarette

Figure I shows the distribution of menthol between the tobacco column and the filter for cigarettes T and F. Menthol migrates from the filter of cigarette F to the tobacco and from the tobacco of cigarette T to the filter. The menthol distribution between the tobacco and the filter of cigarette T (menthol applied to the tobacco) was 95:5 for a 3-day old cigarette and 80:20 for a 28-day-old cigarette. For cigarette F (menthol applied to the filter), the distribution was 30:70 for a 3-day-old cigarette and 59:41 for a 28-day-old cigarette. During 28 days of storage, the amount of menthol on the tobacco of cigarette F and on the filter of cigarette T increased; this continuous increase indicates that the menthol distribution between tobacco and filter does not reach equilibrium in 28 days. From extrapolation of these curves, it appears that equilibrium conditions would be reached after approximately 50 to 60 days. At equilibrium conditions, 70% of the menthol would probably be on the tobacco column and 30% on the filter.

FIGURE I

MIGRATION OF MENTHOL WITHIN A MENTHOLATED CIGARETTE

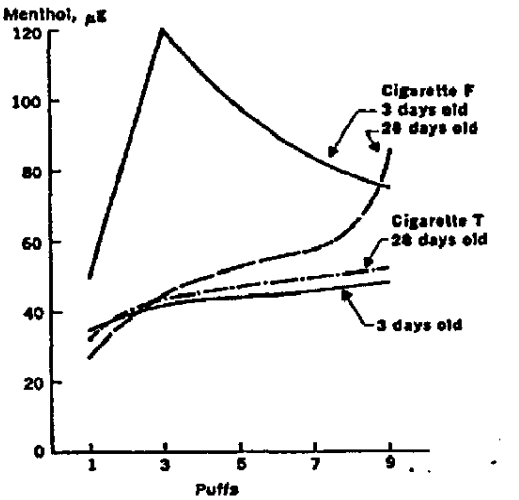


The Effect of Cigarette Age and the Initial Placement of Menthol on Menthol Delivery

Figure II shows that aging cigarette T for 28 days did not affect the amount of menthol delivered by individual puffs, and that the amount delivered by a single puff increased slightly as the number of puffs increased. The age of cigarette F drastically affected the amount of menthol delivered by individual puffs. The amount delivered by individual puffs of a 3-day-old cigarette F increased rapidly during puffs 1-3 and decreased rapidly during the remaining puffs. During puffs 1-6 of a 28-day-old cigarette F, the amount of menthol delivered by individual puffs approximated that of cigarette T, but during the remaining puffs, it increased rapidly.

FIGURE II

EFFECT OF MENTHOL PLACEMENT AND AGE ON MENTHOL DELIVERED DURING SMOKING



Figures I and II show that menthol delivered by individual puffs is not affected when at least 80% of the menthol remains on the tobacco column, but it is affected when only 60% of the menthol remains on the tobacco column.

The data in Table III show cigarette T delivered approximately the same percentage of its menthol after 28 days of storage as it did after 3 days of storage. Cigarette F had a large difference in the percentage of menthol it delivered after 28 days of storage and after 3 days of storage. After 28 days of storage, a larger percentage of menthol was delivered from cigarette F than from cigarette T. Thus, menthol was delivered more efficiently from a mentholated cigarette which had the menthol applied to the filter instead of the tobacco. This type of mentholated cigarette is not practical, however, owing to the large increase in menthol delivered by the last puffs. Therefore, when the amount of menthol delivered per puff and the efficiency of menthol delivery are considered, a more satisfactory cigarette might be one having 70% of the menthol on the tobacco column and 30% of the menthol on the filter.

TABLE III

EFFECT OF CIGARETTE AGE AND MENTHOL PLACEMENT ON MENTHOL DELIVERY

Cigarette Age, Days	Menthol Delivered			
	Cigarette F[a]		Cigarette T[b]	
	mg	%	mg	%
3	0.92	49	0.48	20
28	0.59	31	0.45	19

[a]Contained 1.88 mg of menthol.
[b]Contained 2.34 mg of menthol.

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SUMMARY

Two types of fresh mentholated cigarettes (menthol applied to either the filter or the tobacco) were compared. After 28 days, 20% of the menthol had migrated from the tobacco of cigarette T to its filter, and 41% of the menthol had migrated from the filter of cigarette F to its tobacco. The menthol distribution between tobacco and filter does not affect the amount of menthol delivered by individual puffs over the 95:5-80:20 range but does affect it over the 30:70-59-41 range.

A mentholated cigarette which had menthol applied to the filter delivered menthol more efficiently than one that had menthol applied to the tobacco. Where only the amount of menthol delivered per puff and the efficiency of menthol delivery are considered, a mentholated cigarette with 70% of the menthol on the tobacco column and 30% of the menthol on the filter, might be more satisfactory than a mentholated cigarette which had the menthol applied only to the tobacco.

1. M. P. Newell, P. H. Latimer and L. R. Haefele, 22nd Tobacco Chemists Research Conference, October, 1968; CORESTA Information Bulletin, 1, Abstract 6174 (1969).

2. R. F. Dawson, R. D. Carpenter, F. L. Gager, Jr., R. W. Jenkins, and R. H. Newman, 5th International Tobacco Scientific Congress, 1970; CORESTA Information Bulletin, Special, Abstract B001 (1970).

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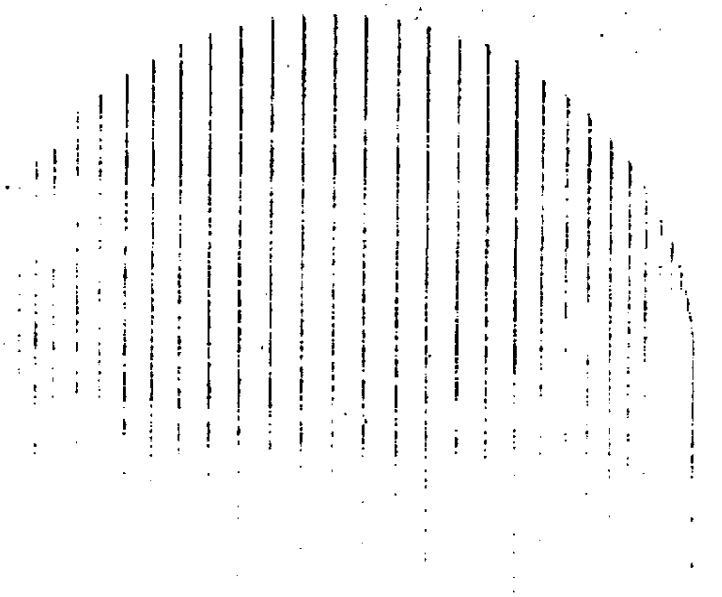

Migration and Delivery of Filter Flavors

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